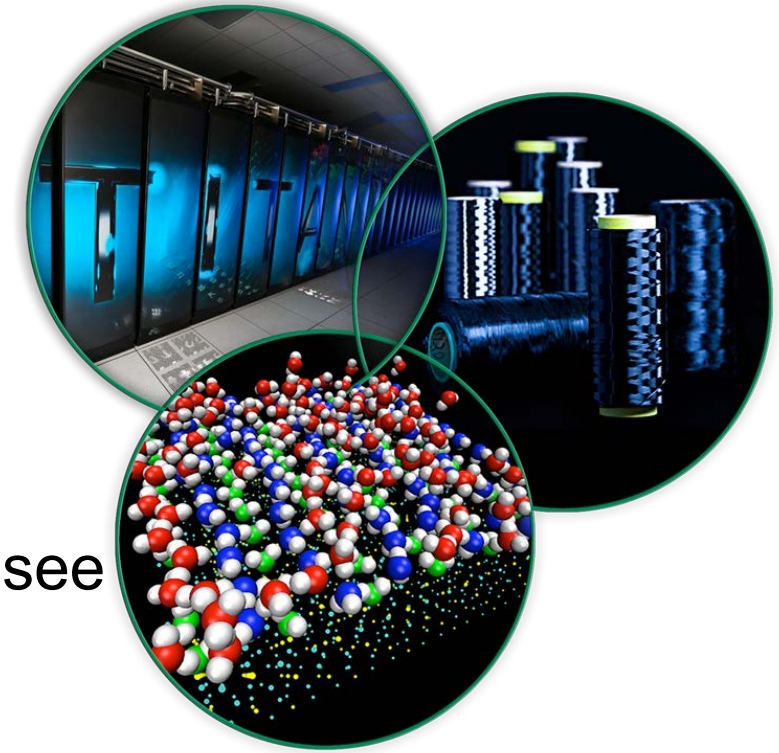
Advanced Carbonization of Low Cost Carbon Fiber



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Project ID #LM069

U.S. DEPARTMENT OF ENERGY

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE U.S. DEPARTMENT OF ENERGY

Overview

Timeline

- April 1, 2010
- September 30, 2013
- ~80% complete

Budget

- Funding received in FY12: $500K from VT/$500K from AMO
- Funding expected for FY13: $350K from VT/$350K from AMO

This project is a portion of an ongoing CRADA with Dow.

Total CRADA funding: $12,760K

- DOE share $5,000K
- Contractor share $7,760K

Advanced Conversion portion of the project is ~60% ($3M) of which VT is co-funding ~$850K with AMO providing balance.

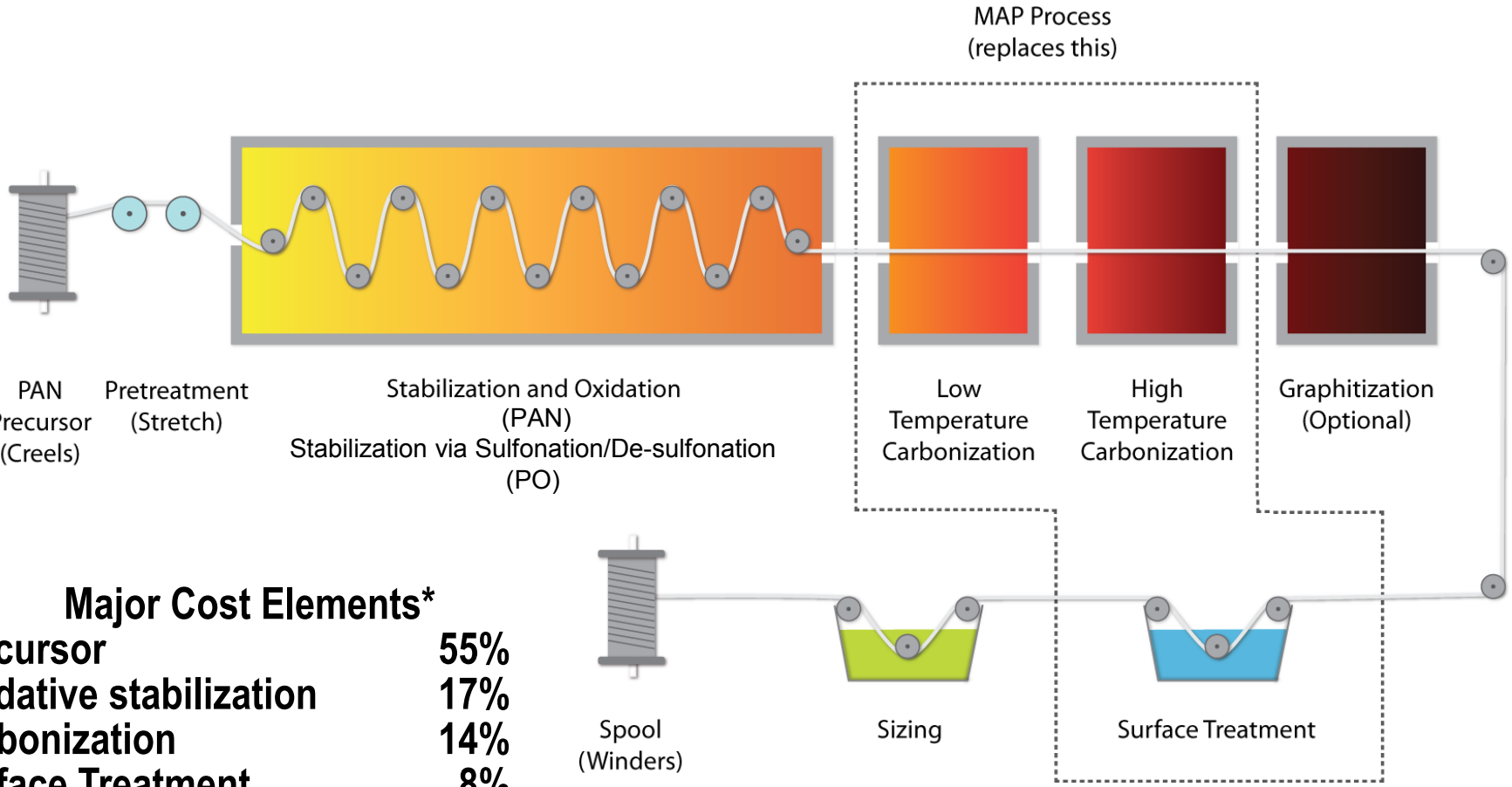
Barriers

- Barriers addressed
  - Carbon Fiber Cost
  - Energy Consumed in Carbon Fiber Manufacturing
  - Flexibility of Carbon Fiber Manufacturing Processes

Partners

- Dow is CRADA partner focused on alternative polyolefinic precursor formulations
- ORNL collaborates on precursor development and conversion of new precursors
- ORNL leads advanced conversion development

Conversion Process Introduction



Major Cost Elements*

Precursor	**55%**
Oxidative stabilization	**17%**
Carbonization	**14%**
Surface Treatment	**8%**
Other	**6%**

*Cost projections from Kline cost model recently updated by ORNL

Advanced Conversion Objectives

Reduce carbon fiber manufacturing costs

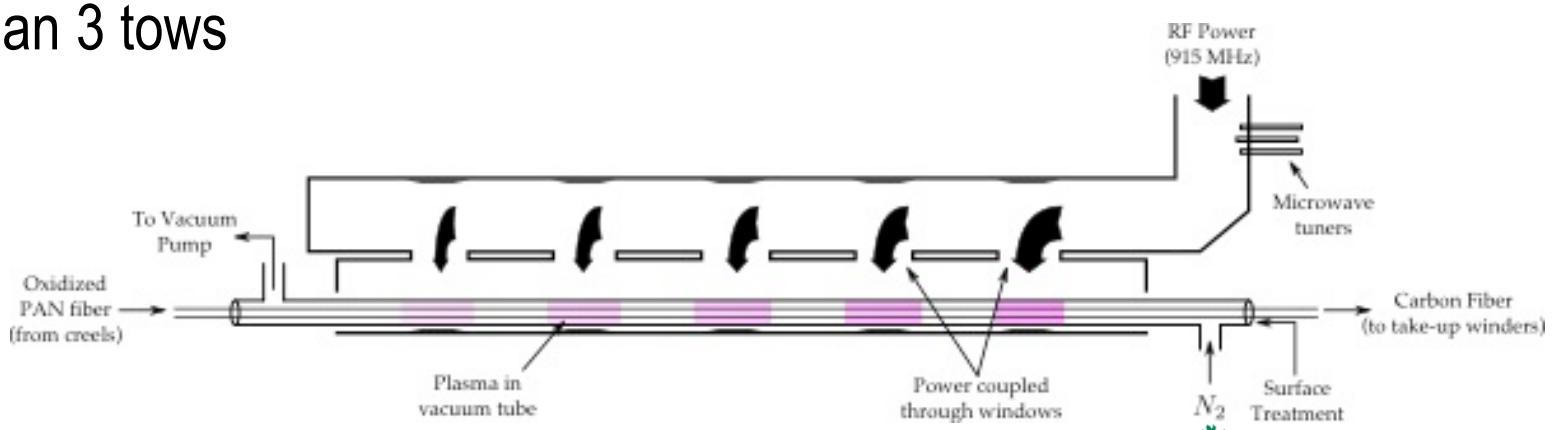
- Scale energy-efficient advanced conversion technology for carbon fibers that are technically and economically viable in transportation (and other energy missions to facilitate commercialization)
- Focus on plasma-based processes applicable to PAN, PO, and other precursors
- Develop and demonstrate equipment and process knowledge adequate for scale-up of an advanced carbonization process
- Deliver specifications for pre-production scale demonstration unit/module

Background

- In previous work funded by the Vehicle Technologies Program, ORNL had demonstrated bench-scale microwave-assisted-plasma (MAP) carbonization of 1-3 tows

however

- System was not robust allowing only short-term operation (~1 hour)
- Sealing and other configuration issues hampered uniformity and operability
- Scale-up will require demonstration of capability to employ modules larger than 3 tows



Major Milestones

Task/ Milestone Number	Title or Brief Description	Task/Milestone Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
MS 1	Formal agreements between Dow and ORNL to delineate project goals, metrics, and responsibilities while facilitating our technical collaboration and protecting intellectual property will be established	9/30/2010		10/31/10	100%	Completed - CRADA signing completed in October 2010.
MS 3	Dielectric measurement system capability installed and initial microwave model completed.	12/31/2010		12/31/2010	100%	Completed - initial measurements taken and data incorporated in modeling effort.

Major Milestones (cont)

Task/ Milestone Number	Title or Brief Description	Task/Milestone Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
MS 5	Complete initial feasibility determination for atmospheric plasma carbonization or plasma torch alternative.	6/30/2011	9/30/2011	9/30/2011	100%	Feasibility assessment has been completed on initial "Surfaguide" approach; conclusion is that this approach is not practical. Contract to evaluate alternative atmospheric glow plasma technology is in progress.
MS 6	Establish energy balance for single and multiple tows	9/30/2011		9/30/2011	100%	Initial energy balance completed. This data is being used to guide further process economic concentration and will be updated as appropriate.

Major Milestones (cont)

Task/ Milestone Number	Title or Brief Description	Task/Milestone Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
MS 9	Establish design parameters for one vs. two chamber plasma reactor for final project demonstration module(s), establish energy balance for atmospheric plasma carbonization process (if feasible) and update energy balance for MAP process.	9/30/2012	6/30//2013		70%	Milestone delayed to capture results of related equipment developments.
MS 10	Attain 25 Msi (170 GPa) tensile modulus and 250 ksi (1.70 GPa) tensile strength for lead precursor candidate in a carbon fiber.* *	3/31/2013		3/31/2013	100%	Properties met with selected Dow formulation.
MS 12	Complete design specification for an advanced carbonization module based on plasma processing at 25-ton-per-year production levels for replacement of conventional carbonization unit in ORNL's Carbon Fiber Demonstration Facility scheduled for completion in FY 2013.	9/30/2013				On schedule

Technical Approach

- *Task 1. Develop and/or evaluate alternative approaches sealing approaches, reactor materials, and related hardware to enable continuous operation >8hrs.*
- *Task 2. Develop and evaluate atmospheric pressure solutions to mitigate or eliminate sub-atmospheric pressure processing .*
- *Task 3. System modeling of* deposition and plasma intensity implemented to guide hardware design and process development.
- *Task 4. Scale to ≥ five large tows while meeting property targets of 250 ksi and 25 Msi.*

Technical Approach (cont)

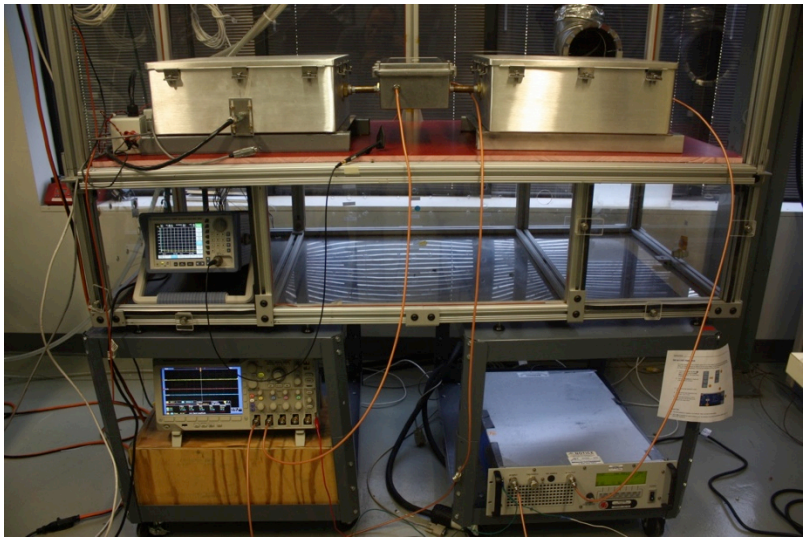
- *Task 5. Determine preferred microwave/plasma parameters and profiles necessary to minimize residence time (<3 minutes); Estimate energy requirement at various scales.*
- *Task 6. Determine long-term, continuous operability utilizing 4-8-hr runs to extrapolate stability of operations at >100 hrs.*
- *Task 7. Develop information necessary to generate specification for a design of a robust advanced technology carbonization unit to be built and operated in the ORNL Carbon Fiber Technology Facility*.

Atmospheric Plasma Investigation

- Atmospheric plasma could potentially replace portions or augment the MAP process. By operating in open atmosphere, vacuum/sealing and potentially other hardware issues are minimized.
- Present work focusing on near-field capacitively coupled RF plasma sourcing integrated with a direct electrical heating technique. Investigation currently at bench-top feasibility stage.

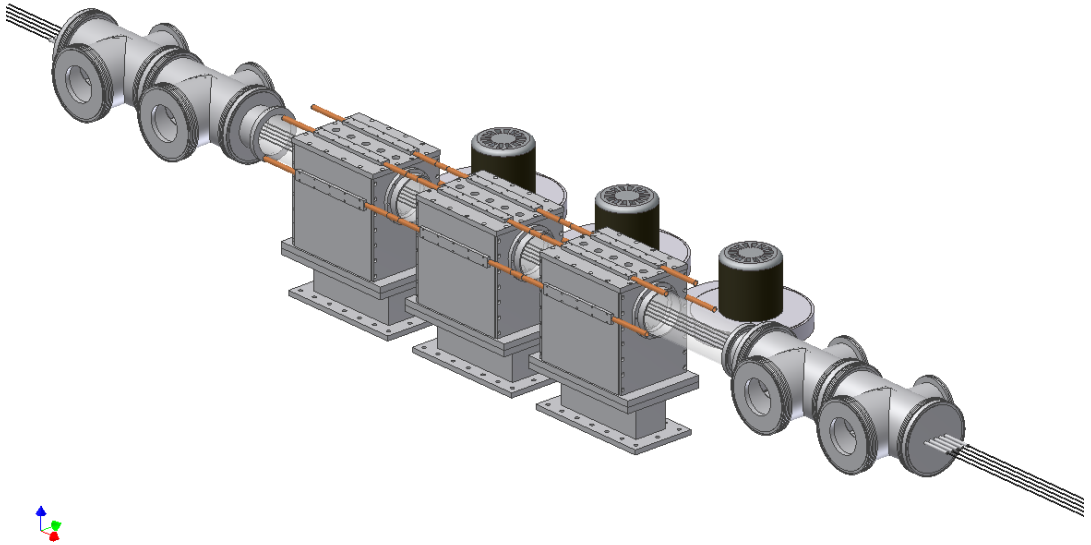
System Facts:

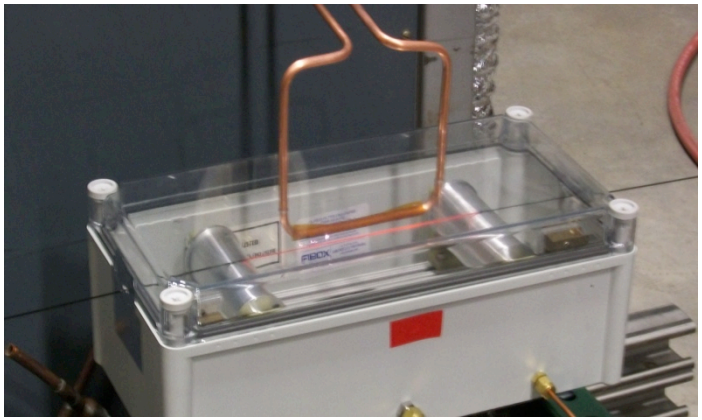
- Single tow batch process
- Hybrid electrodes accomplish heating and plasma generation.
- Present scale consumes ~100W for one fiber tow



Energy Deposition Options

- To maximize efficiency and economics, we continue to consider single vs dual chamber (i.e. LT/HT or pre-heat/primary heat zone) approaches



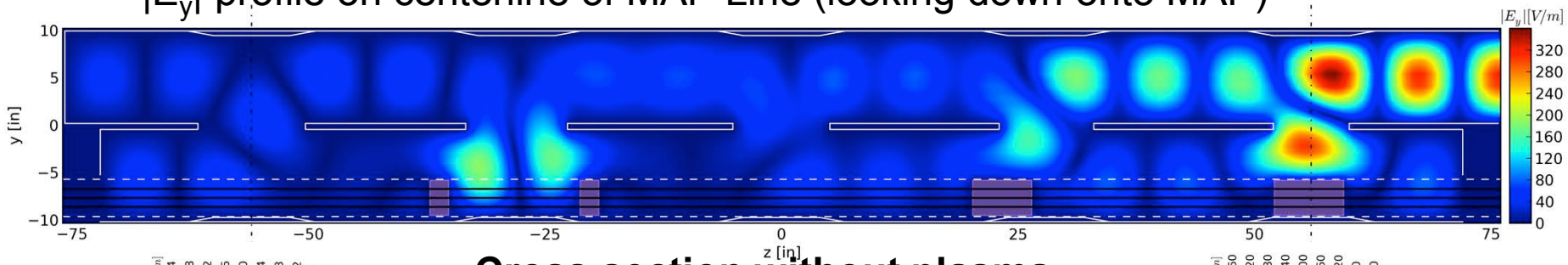


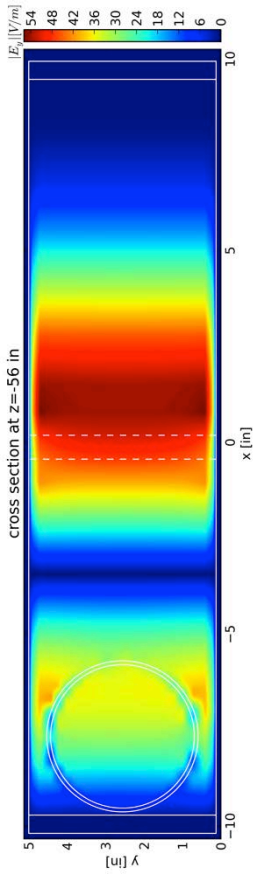
Induction Preheat Antenna

- Experiments are planned to determine if a multi-zone microwave applicator approach for enhancing energy distribution control along the chamber length improves tow-to-tow energy distribution.

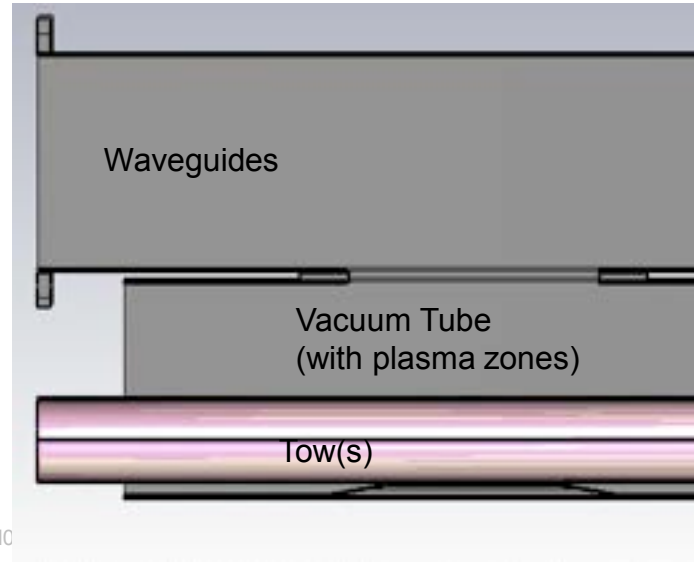
Modeling of the MAP Line

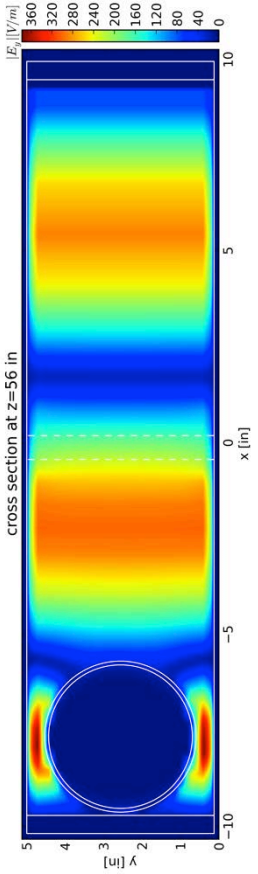
$|E_y|$ profile on centerline of MAP Line (looking down onto MAP)





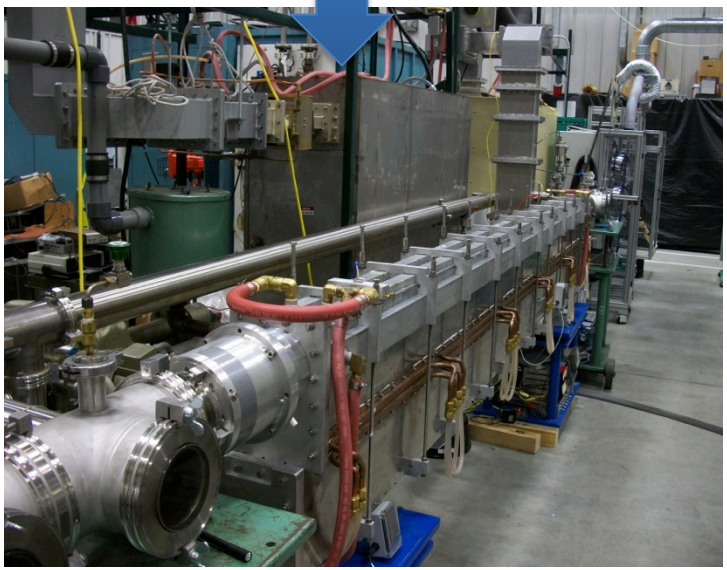
- **Cross section without plasma shows electric fields permeating vacuum tube.**
- **Cross section with plasma shows shielding of fields in plasma and high fields above and below. Experiment corroborates.**





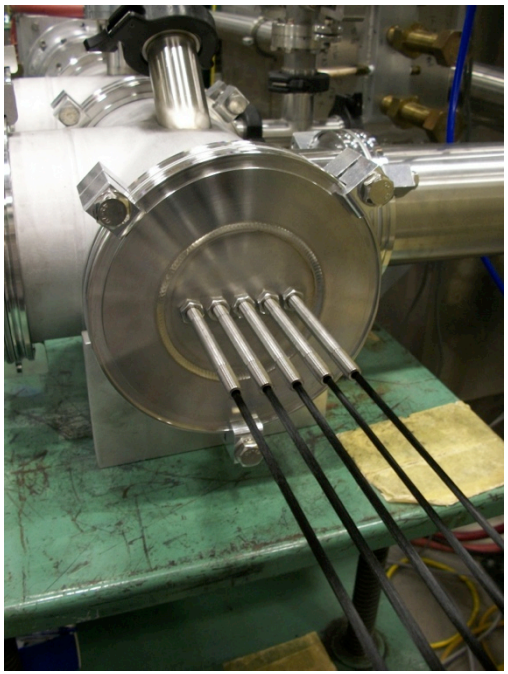
Advanced System Developments

- Microwave energy supply rotated from horizontal to improve distribution uniformity
- Significantly larger processing chamber implemented for 5 tows
- 5 tow fiber handling system fabricated, installed, and operational



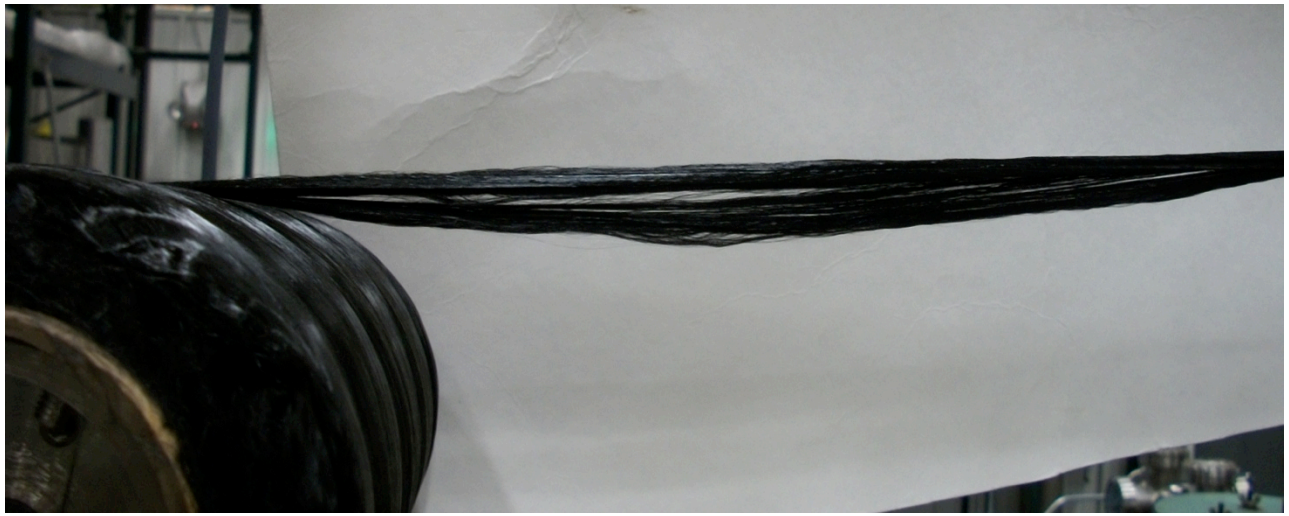
System Developments (cont)

- New tube and tow seal approaches conceived and demonstrated to improve durability
- Tow orientation changes modeled and evaluated
- Continuing improvements in effluent handling are being demonstrated

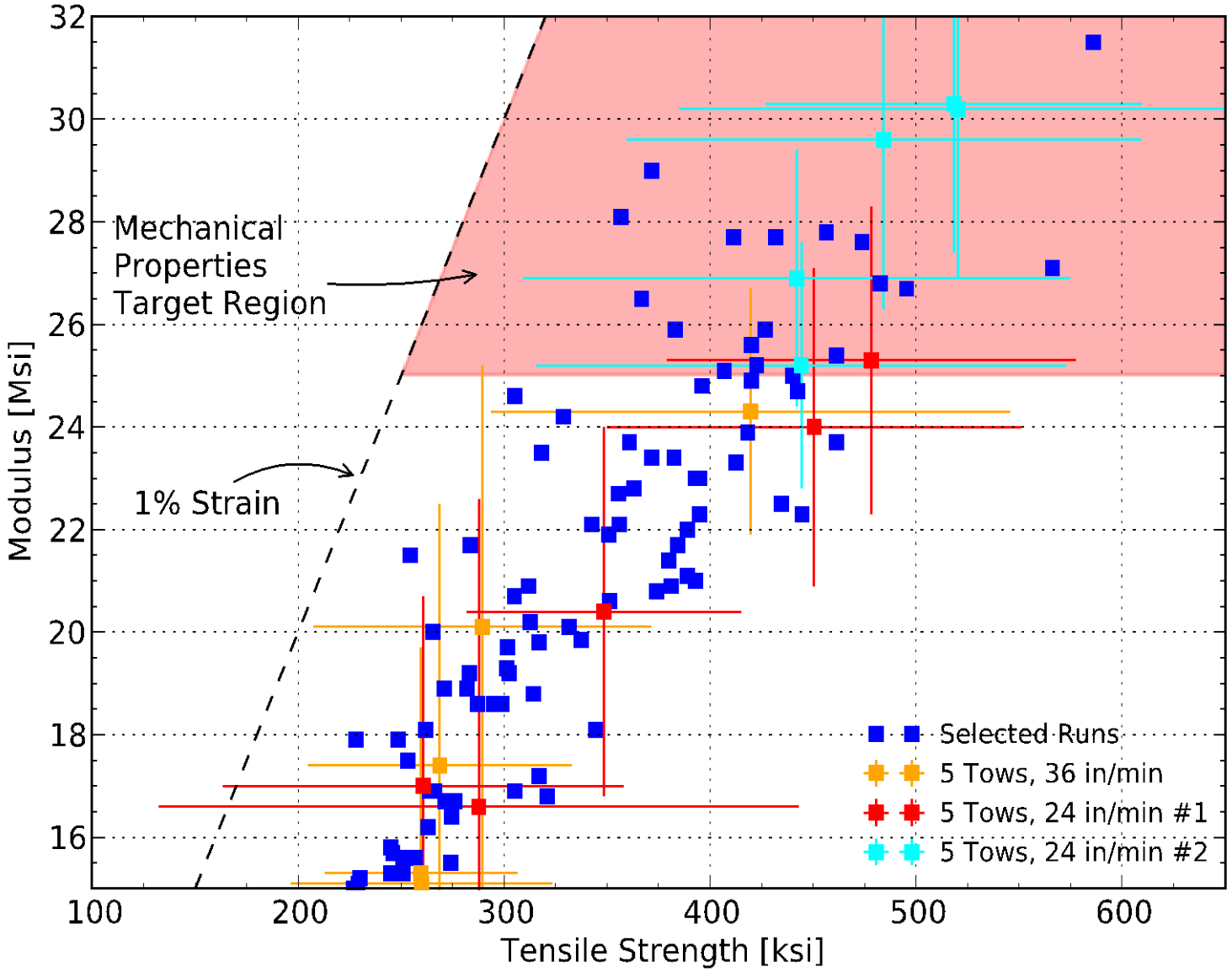


Recent Results are Promising

- Demonstrated stable system in 8 hour continuous operation with 3 tows
- 5 tow scaling is going well
- Energy deposition and data uniformity is improving in spite of feedstock (oxidized PAN) tension non-uniformities



Recent Data Meets Target Levels



Advanced Carbonization Economic Model* Summary

	Conventional Technology	Advanced Carbonization	Savings
1500 t/y Scale	$10.20	$8.89	$1.31 (13%)

- Low Temp. (LT), High Temp. (HT), and surface treatment functions performed by a single, lower cost microwave assisted plasma (MAP) unit
- For the same commodity PAN precursor, total material costs are essentially the same for MAP carbonization vs. conventional carbonization
- Cost savings driven by substantially reduced carbonization, abatement, and surface treatment processing costs
- Conventional total processing costs (capital, labor, energy) $5.18/lb versus $3.93/lb for MAP. $8.15/lb under high production volume (18,000 t/y)

** Model developed by S. Das and J. Warren for DOE/VT in FY2012.*

Collaboration

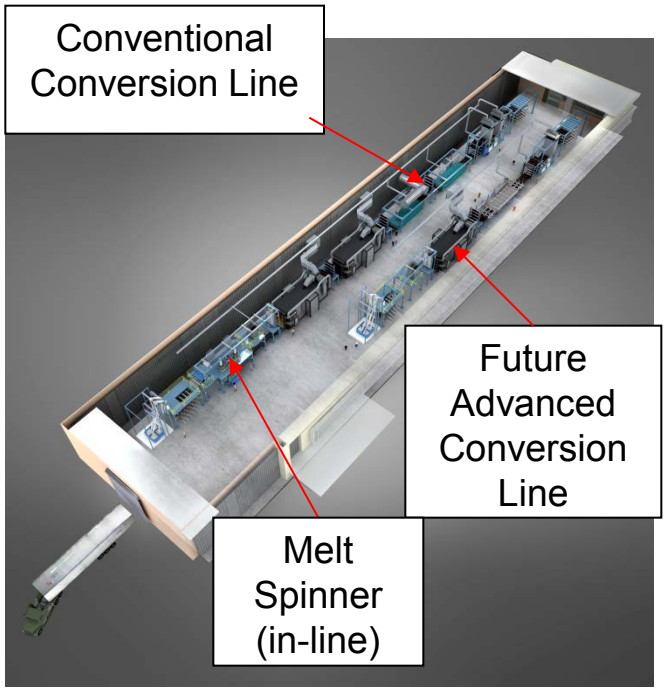
- VTO and AMO are jointly funding Advanced Conversion development
- Dow is CRADA partner on overall Alternative Carbon Fiber Precursors and Conversion Technologies project
  - Dow provides alternative PO-based precursor formulations and collaborates with ORNL on development of associated conversion processes
  - ORNL leads work in Advanced Conversion processes concentrating on cost and energy savings in carbonization utilizing plasma-based processes applicable to PO, PAN,and other precursors
- ReMaxCo is developing and evaluating atmospheric plasma-based processes similar to related oxidation project, but requiring modified approaches
- Various existing carbon fiber equipment and carbon fiber product manufacturers and other interested parties have approached ORNL with interest in licensing and/or future collaboration
- Plans are to more formally assess interest and investment options for next phase in Q4 of FY2013 when approach is better demonstrated.

Future Work

- Resolve feasibility of atmospheric plasma process for all or portions of carbonization
- Demonstrate enhanced energy distribution with multi-zone microwave applicator
- Complete fully integrated advanced carbonization system and demonstrate technical capacity and projected economics for scale-up
- Demonstrate applicability to PO-based precursor
- Complete specifications for CFTF scale demo unit
- Develop potential collaborative partnership opportunities plans for next phase

Pathway to Commercialization

- In parallel initiative, new ORNL Carbon Fiber Technology Facility (CFTF) is now open
  - Specifically focused on demonstration and scale-up (25 tons/year) of emerging technologies for large-scale material and process evaluations
  - Maximum flexibility is being incorporated to handle alternative precursors in wide variety of formats in conventional conversion processes
  - Space is allocated for advanced conversion processes that could be interleaved with conventional processes



Summary

- Team is making substantial advances with MAP technology in demonstrating improved operability, more practical hardware, and ability to scale to more tows
- Further improvements are being investigated to improve uniformity of energy deposition to get tow-to-tow variability <10% and including alternative approaches for portions of the carbonization process to enhance economic feasibility
- Plans in place to complete project by
  - Fully integrating advanced carbonization system and demonstrate technical capacity and projected economics for scale-up
  - Demonstrating applicability to PO-based precursor
  - Completing specifications for pre-production (CFTF, i.e. 25-ton-/year) scale demo unit to facilitate commercialization interest/commitment
  - Developing potential collaborative partnership opportunities plans for next phase

Summary of Key Criteria

Relevance

- Opportunity for significant cost reduction in conversion of carbon fiber

Approach/Strategy

- Builds on small-scale technology demonstrator and addresses key issues in scaling

Technical Accomplishments

- Advances achieved in process durability and scaling while meeting property goals

Collaboration

- Part of ongoing CRADA with Dow, teaming with internal and external SMEs, and planning for scaling partners

Future Work

- Completion and demonstration of integrated module
- Development of partnerships and plans for pre-production scale